

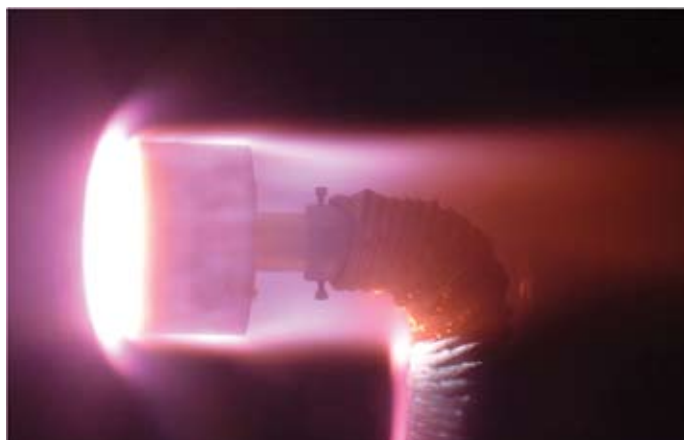
## Advanced Thermal Protection Systems for Hypersonic Vehicles

New missions for hypersonic vehicles impose demanding performance (200 W/cm<sup>2</sup> maximum heat flux, 2000°C maximum temperature, 30 minute atmospheric flight), mass, and volume requirements on the thermal protection systems (TPS) that cannot be met using existing materials. The Organic Materials Department is providing materials expertise to programs developing new TPSs for hypersonic vehicles.

Carbon-carbon composites were chosen as the base material for these systems for their exceptional mechanical properties at high temperatures. The major disadvantage of carbon-carbon composites is their susceptibility to oxidation at temperatures as low as 500°C. To address this problem, the Organic Materials Department has developed oxidation protection treatments using polydimethylsiloxane infiltrants and siloxane-based coatings. During flight, these materials decompose to form oxidation-resistant silicon glasses on the surface of the carbon-carbon, protecting it from oxidation. In-situ formation of the protective coating minimizes problems due to the mismatch in coefficient of thermal expansion between the carbon-carbon substrate and the glass coating.

Preliminary evaluation of materials was performed using laboratory ovens. However, to evaluate materials under conditions (heat fluxes, heating rates, and temperatures) representative of a flight environ-

ment, specialized test equipment is required. To address this need, new test fixtures were constructed at the National Solar Thermal Test Facility solar tower for long duration, high heat flux testing. In addition, samples were



**Sample under test using the 20 MW Aerodynamic Heating Facility arc-jet at NASA Ames Research Center**



**Solar tower test underway**

recently tested using the 20 MW Aerodynamic Heating Facility arc-jet at NASA Ames Research Center.

For a ten-minute solar tower test with a heat flux of 200 W/cm<sup>2</sup>, the best performing oxidation protection schemes reduced the mass loss from 70-80% for unprotected carbon-carbon to 20%. Ongoing research will focus on understanding the mechanism of oxidation protection to enable optimization of the treatment material, developing improved processing methods to enhance the performance of the oxidation protection scheme, and understanding the effect of the carbon-carbon structure on the TPS performance.

This work was funded in FY05 by the Prompt Global Response Grand Challenge LDRD and will be funded by a new LDRD in FY06 and FY07.

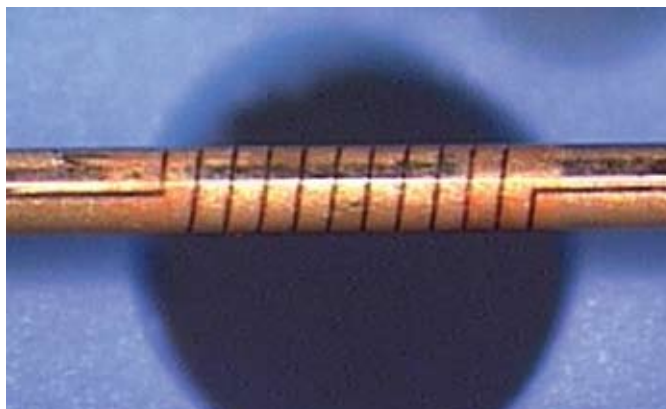
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## Tech Updates

### How Low Can You Go? $\mu$ -NMR Tubes Packaged in LTCC

**N**uclear Magnetic Resonance (NMR) is well known for chemical analysis, including medical applications. In some applications, however, the volume of the sample to be analyzed is extremely small (one millionth to tens of billionths of a liter). The signal-to-noise ratio depends on the volume of the sample. Smaller Micro-Nuclear Magnetic Resonance ( $\mu$ -NMR) tubes can be used to efficiently increase resolution on small samples by many times.

How small can these tubes be? Researchers at Sandia are answering that question using a focused ion beam (FIB) and the results are encouraging (T. Alam, Chemical Analysis and Remote Sensing and D.P. Adams, Thin Film, Vacuum and Packaging). Starting with hollow glass tubes that are slightly larger than a human hair, a thin metallization layer is applied to a key portion of the tube. A special holder is used in the FIB to manipulate the tubes and use gallium ions to mill tiny cuts into the metallization as the tube is rotated. The resulting coil meets a critical trade-off, having a small size with an adequate number of turns, yet maintaining a low



**This hollow glass tube is thinner than a human hair. Using focused ion beams and gallium ions, features are milled from a thin metallized coating.**

resistance. All this is accomplished on tubes that are extremely fragile and can be easily broken in handling.

The tubes have been 'packaged' in Low Temperature Cofired Ceramic (LTCC) due to a capability for electrical connections and a newly developed capability for convenient microfluidic channels. The path lengths from the tube connections to controlled impedance lines must also be short. The results are that resolutions are achieved that outpace any prior result. Attempts are underway to fur-

ther reduce the dimensions of this device so to make it compatible with typical electronic board applications and increase sensitivity beyond current levels. Near-term plans include the pre-packaging of the tubes, so that they see handling after they are already in a rugged protective

package. Commercial microfluidic nanoports complete the assembly and its low profile permits it to be placed into a strong magnetic field for testing. The work is proceeding with LDRD funding. Manufacturing technology to place these tubes in routine use will greatly improve the quick analysis of critical samples for the nation.

The project involves T. Alam (Chemical Analysis and Remote Sensing), D.P. Adams, K. Peterson, M.J. Vatile, V.C. Hodges, A. Fresquez, C. Benally, G. Schwartz (all in Thin Film, Vacuum and Packaging), and R. Torres (Ceramic and Glass Processing).

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### Application of Low Temperature Co-Fired Ceramic For Neutron Tube Components

**T**he potential for enhanced performance and size reduction of next generation neutron tubes can be realized using Low Temperature Co-fired Ceramic (LTCC) technology. This design approach has been successfully demonstrated in collaborative effort by Fernando Uribe and Chuck Walker, both of 02452; Scott Reed of 02454; and Dan Krueger of HFM&T in Kansas City.

Neutron tube architecture dictates the need for precision target resistors capable of handling high-energy pulses. The use of wire wound (historically) and thick film resistor technology (more recently) has been demonstrated to successfully meet

these requirements. The disadvantages of these two approaches are the inherent increase in assembly complexity, as well as the need for hermetic feed-through in the neutron tube ceramic target insulator. The potential for integration of the target resistor into the insulator body of the neutron tube will inherently reduce the neutron tube parts count and assembly complexity.

The design concept is based on fabrication of the neutron tube target insulator body using DuPont green tape. The target resistor is patterned, using screen-printing techniques, on the internal tape layers of the LTCC insulator body. The layers of green

tape are laminated and sintered to form a monolithic plate type structure. Due to the inherent complex geometries of the NT target insulator, processes were developed to machine the LTCC structure.

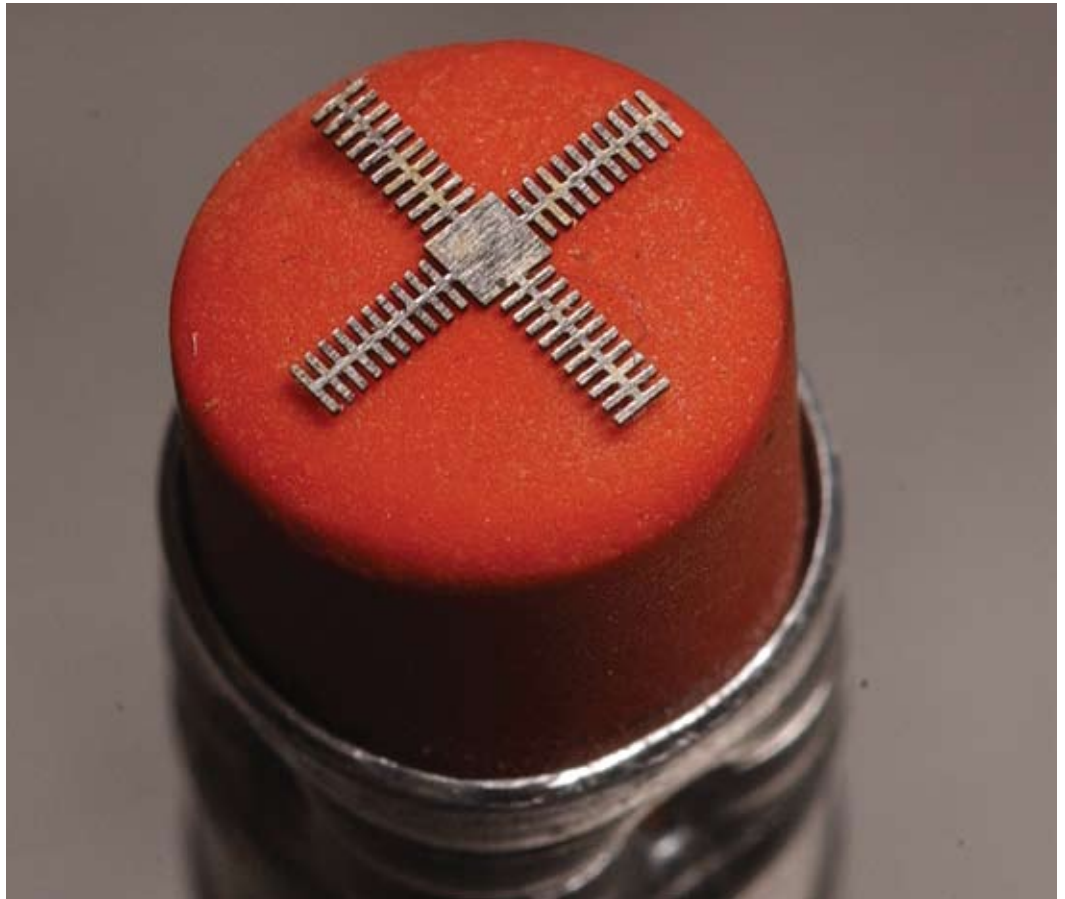
Since the resistor is embedded in the insulator body, conventional laser trim techniques to adjust resistance are not viable. Post-sintering thermal treatment techniques were therefore developed that allow adjustment of resistance to the desired target value. Neutron tube assembly architecture necessitates brazing of the ceramic insulator to the Kovar target and base plate. Materials and braze processes compatible with LTCC were identified. Survivability of the LTCC integrated

**(Continued, Co-fired, next page)**

## Sandia's Precision Meso Manufacturing Program Has a New Focus

**M**any engineers and product realization teams at Sandia National Laboratories are currently engaged in efforts to create revolutionary national security products that feature unprecedented functionality in ever-smaller, more portable configurations. In the course of development, the Sandia technology community has realized the need for manufacturing capabilities that expand upon what traditional microfabrication provides. Often the need arises when functional specifications call for very small parts from materials (such as metals) that are currently incompatible with lithography, wet etching, or deep reactive ion etching (DRIE) techniques. This situation is particularly evident in micromechanical surety devices that must reliably transmit relatively high forces in harsh environments. More compelling is the expanding class of microfluidic products that incorporate complex, three-dimensional, miniature fluid handling systems (pumps, valves, etc.). From these aggressive new product requirements, Sandia's thrust in precision meso manufacturing has arisen.

The term "meso," derived from the



**A mesoscale test article created by femtosecond laser machining**

Greek mesos, meaning "intermediate" or "in the middle," describes operations on a length scale ranging from hundreds of micrometers to one centimeter. One looks no further than his or her own wristwatch or the kids' toy train to realize that the foundation for precision meso manufacturing has existed for centuries. Sandia's

program takes off from there. Meso manufacturing at Sandia employs an updated suite of innovative fabrication and metrology tools that complement each other to make advanced meso products a reality. The meso manufacturing suite includes micro-wire EDM, diamond turning/ultra-high precision machining, femtosecond laser machining, and advanced metrology. If you would like to learn more about precision meso manufacturing and how it can improve your product, contact Jeremy Palmer (505-284-9623, [japalme@sandia.gov](mailto:japalme@sandia.gov)) (femtosecond laser and high resolution stereolithography); David Gill (505-844-1524, [ddgill@sandia.gov](mailto:ddgill@sandia.gov)) ( $\mu$ EDM and diamond turning); or Andre Claudet (505-845-1381, [aaclaud@sandia.gov](mailto:aaclaud@sandia.gov)) (metrology for precision meso manufacturing). You'll never look at your watch the same way again!

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### Continued, Co-fired

resistor to high energy pulses was demonstrated and characterized. Ultimately test devices resembling neutron tube configuration were assembled and tested after exposure to adverse temperature environments. Early failures indicated low strength between the LTCC integrated resistor and the Kovar piece parts. However, optimization of the braze process has resulted in enhanced joint strength.

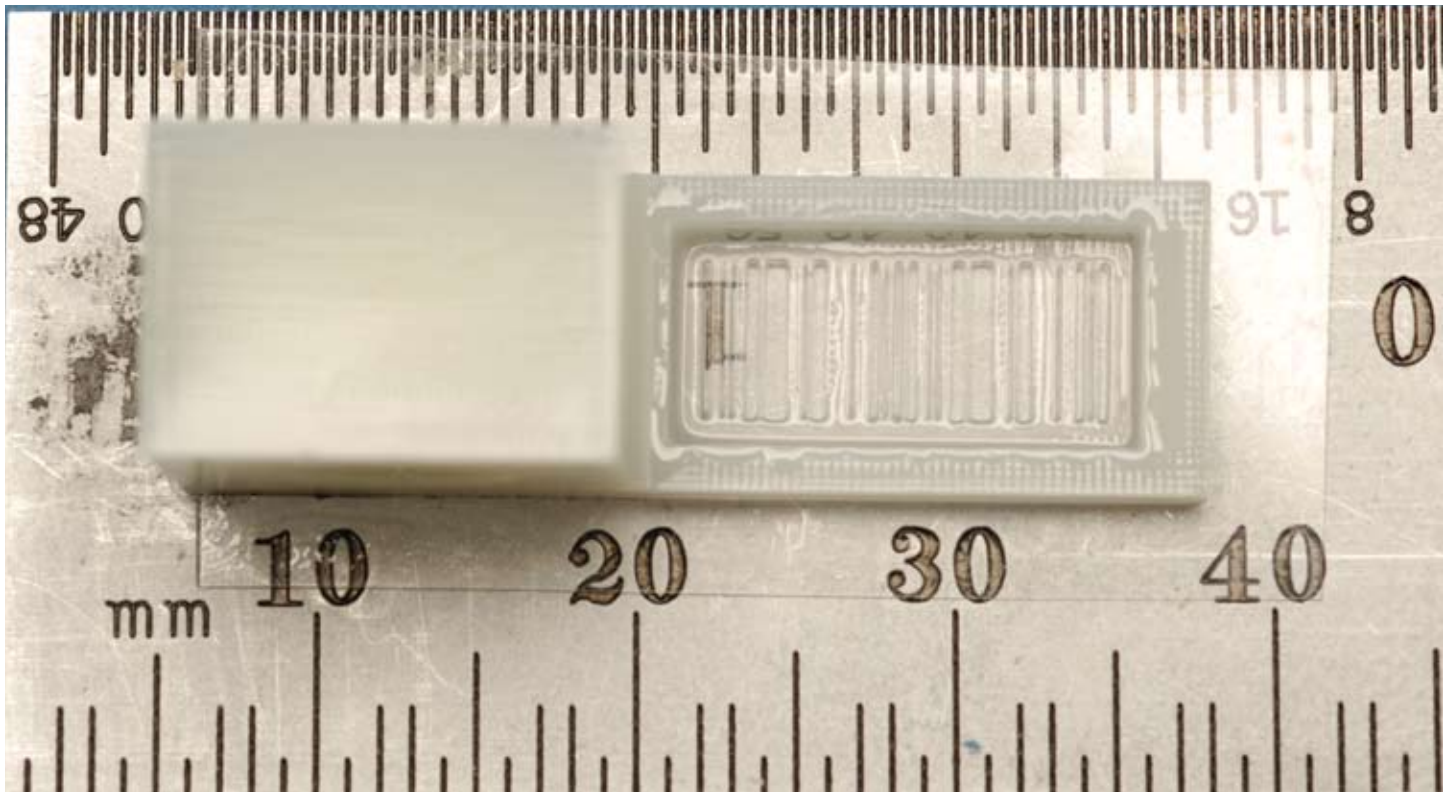
In conclusion, LTCC is a technology

that offers the potential for integration of the target resistor into the interior of the neutron tube thereby reducing part count, size, hermetic feed-through and, ultimately, assembly complexity of next generation neutron tube designs.

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## Hadamard Masks Made With Microstereolithography



A Hadamard mask created by microstereolithography

Through the use of modern microstereolithography technology, a form of additive rapid prototyping, optics developers have the freedom to integrate mounting and positioning structures directly into an optical mask assembly. Microstereolithography is an additive process in which solid objects are built out of UV curable liquid polymer. This polymer solidifies as the UV laser draws out the first layer (100 $\mu$ m) of the object. The platform then moves down 100 $\mu$ m, and the laser draws the second layer of the part.

An object is built layer by layer from the bottom up, therefore having the ability to take on any shape. This additive layer process also allows other items to be integrated into the object by stopping the build halfway through, placing the item, and then continuing the

building process around the item placed. This eliminates the need for assembly and fasteners.

Through the use of the Sony SCS 6000 stereolithography apparatus (SLA), feature sizes as small as 75 $\mu$ m and larger than 300mm can be created in the same build. Students have designed and built a Hadamard optical mask using microstereolithography that integrates the mounting structure directly onto the mask. Hadamard masks are used in spectrometry to create a defined signal with a high signal to noise ratio (SNR). The Hadamard mask being created has slits and solidified strips of 150 $\mu$ m or larger (see figure). Due to laser over cure and other parameters, adjustments were made iteration by iteration until appropriate mask tolerances were met. At the CAD level, the mounting structure

and/or the mask can be modified to meet specific needs. A conceptual design of an entire positioning system that integrates all the components necessary for one compact optical spectrometry system created with SLA is underway.

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*The Manufacturing Science & Technology Quarterly* is produced by a rotating team of Center employees.

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## Mfg. S&T Successful in LDRD Proposals

The Manufacturing Science and Technology Center strives to fulfill its mission to “develop and apply advanced manufacturing processes” through many different means, one of the most important being Laboratory Directed Research and Development (LDRD) projects. These projects offer the opportunity to greatly increase our understanding of new processes and to promote significant collaboration with other centers in the Laboratories. This year, Center personnel were awarded five new-start LDRD’s.

1. Principal Investigator (PI) Jeff Galloway of the Organic Materials Department (02453) and Project Manager (PM) Mike Macha (05422) were awarded an LDRD entitled “Development and Optimization of Thermal Protection Materials for Hypersonic Vehicles.” This project is jointly sponsored between the Advanced Manufacturing, Military Technologies and Applications, Engineering Sciences, and Materials Science and Technology investment areas. The team (organizations 02453, 01815, and 01515) will be studying oxidation protection schemes for carbon-carbon composites to enable their use as heat shields on advanced hypersonic vehicles.

2. PI Pin Yang and PM Tim Gardner of the Ceramic and Glass Processing Department (02454) were awarded an LDRD entitled “Low Cost, Meso Scale Parts Fabricated from Nanocrystalline Materials.” The project team (02454, 02455, 01647, and 01813) will create nanocrystalline bulk material through low-temperature densification and will study the material properties, machinability, and utilization of these materials for meso scale components such as

surety mechanisms.

3. PI Chris DiAntonio and PM Tim Gardner of the Ceramic and Glass Processing Department (02454) were awarded an LDRD entitled “Advanced Manufacturing of a Novel Functional Material.” The prevailing target of this LDRD is to develop an advanced, low-cost manufacturing process that results in a lead-free ferroelectric with comparable properties to the presently used lead-based compositions (PZT and derivatives). The team includes organizations 02454, 01815, 01816, 01822, and 01130.

4. PI David Gill of the Mesoscale Manufacturing and System Development Department (02455) and PM Tom Voth (01433) were awarded an LDRD entitled “Titanium Cholla – Optimized, Lightweight, High-Strength Structures for Aerospace Applications.” The team (02455, 01433, 01435, and the University of Rhode Island) will investigate the novel methods of optimization of 3-D structures for specific loading applications and then develop the methods to make these highly complex structures using layer additive manufacturing methods.

5. PI David Gill of the Mesoscale Manufacturing and System Development Department (02455) and PM John Smugeresky (08772) were awarded an LDRD entitled “New, Low-Cost Material Development Technique for Advanced Rapid Prototyping.” The team (02455, 08772, 01814) will utilize computer modeling and the Laser Engineered Net Shaping™ (LENS®) machine to develop methods of small-scale prototyping of novel metal alloys with unique properties.

## Ceramic & Glass Processing Green Belt Council

Ceramic & Glass Processing (2454) has its very own in-house Lean Six Sigma (LSS) greenbelt council. The council was created approximately one year ago, and the mission is to keep Lean Six Sigma alive and functioning in the department and to introduce LSS to other departments. Steve Lockwood is the resident black belt who guides the group, and Margaret Sanchez is the in-house green belt who leads the group. There are four other positions on the council which are rotated at different intervals every six months.

The council aids individuals within the department in becoming green belt certified. Members of the council encourage peers to register for the green belt class (GT 100). After the class has been completed, the council can guide an individual in choosing a project to undertake. The council

is willing to help organize the project and then mentor executing the project for green belt certification. Actually working a project in the LSS fashion aids the individual in becoming familiar with the LSS methodology. Assistance is also available for composing the final presentation after the project has been completed. After becoming a certified green belt, the individual is expected to participate and lead other projects in the department.

The goals set for the Ceramic & Glass Processing department by its manager, Tim Gardner, are to have 70% of the department take the LSS class and become green belt trained and for 50% of the department to be green belt certified. Currently 83% of the department has taken the LSS green belt class and 61% of the department is certified as a LSS green belt. Tim has asked everyone in the department

to participate in at least two LSS events per year or lead one event per year for continued certification.

The council has also introduced the LSS methodology to other departments in the Center. The group has assisted with three different 6S projects in the Bldg. 840 Machine Shop: Wire EDM (190B), Apprentice Shop Phase I (E19), and Apprentice Shop Phase II (E19). In building 878, the council has assisted with 6S projects in labs B1000, B802, B807, B401, B403, B406, B408, B410 in Material Characterization and labs A711A and B1600 in Thin Film, Vacuum and Packaging. The council plans on continuing to help introduce and sustain LSS methodology throughout the center and across Sandia.

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## Manufacturing and Science and Technology Center Holds First Apprenticeship Program Graduation Ceremony

The Manufacturing Science and Technology Center's Advanced Manufacturing Trades Training Program (AMTTP) was certified as a formal apprenticeship program by the New Mexico State Apprenticeship Council on May 20, 2004. Since then, there have been thirty-three graduates. The first annual graduation ceremony and reception for these graduates was held on November 2, 2005, at the Sheraton Uptown. This ceremony was hosted by Juanita Sanchez, the AMTTP Training Coordinator.

More than 35 trade focus sub-committee members, union officers, team leads, managers, and other distinguished guests, including the Director of NM State Apprenticeship Council came out to celebrate the

accomplishments of these graduates who are now classified as Full-Time Employees (Tradesmen). The graduating apprentices have completed more than 7,500 hours of intense hands-on training, tour rotations, and classroom instruction, as well as considerable skill assessments to meet the unique aspects and skill standards for each trade group.

Gil Herrera, Manufacturing S&T Center Director, gave the address and presented certificates of completion from Sandia National Laboratories and the State of New Mexico to the graduates. Congratulations, Graduates, on your hard-won accomplishment!

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**Mechanical Measurements (2433-1):** Barbara Allison, with Gil Herrera (left) and manager Paul McKey.



**Machining (2433-2): (l-r) Michael McReaken, Delvacchio Charley, Reuben M. Baca, Erik Hart, Quy Dinh, manager Paul McKey, and Aaron Otzenberger.**

**At right: Electronic Fabrication (2434-1):** Leonard Dixon, Lin Nguyen, Elizabeth Montgomery, Kevin Santistevan, and Grace Gallegos, with manager Phil Gallegos. Not shown are Isaac Garcia, Kenny Gutierrez, Christian Maestas, and Michael Maness.



**At left: Materials—Thin Film, Vacuum & Packaging (2452): (l-r) James Kuthakun, Richard Sanchez, Troy Gourley, Armando Fresquez, Tim Turner, with manager retired Bob Poole. Not shown is Marlene Chavez.**



**Materials—Active Ceramics (2454):** Warren Lubin, Angel Vega-Prue, Ray Hannah, Brenda Pentecost, Mark Perea, Margaret Sanchez, Nelson Capitan, with retired manager Bob Poole. Not shown are Audrey Gallegos and Tom Chavez.

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